

REPORT DOCUMENTATION PAGE

AFRL-SR-BL-TR-99-

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Public reporting burden for this collection of information is estimated to average 1 hour per response gathering and maintaining the data needed, and completing and reviewing the collection of information, including suggestions for reducing this burden. □ Washington Headquarter Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget

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1. AGENCY USE ONLY (Leave blank)			2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
			Nov 3, 1998	Final Technical Report 15 Feb 97 to 29 Aug 97
4. TITLE AND SUBTITLE			5. FUNDING NUMBERS	
Creep Behavior of Solid Solution Strengthened Y3A15012			F49620-97-1-0097	
6. AUTHOR(S)				
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7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)			8. PERFORMING ORGANIZATION REPORT NUMBER	
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9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)			10. SPONSORING/MONITORING AGENCY REPORT NUMBER	
AFOSR/NA 801 N. Randolph Street, Rm 732 Arlington, VA 22203-1977			F49620-97-1-0097	
11. SUPPLEMENTARY NOTES				
12a. DISTRIBUTION AVAILABILITY STATEMENT			12b. DISTRIBUTION CODE	
Approve for public releases; distribution unlimited.			A	
13. ABSTRACT (Maximum 200 words)				
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14. SUBJECT TERMS			15. NUMBER OF PAGES	
			8	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT		18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
Unclassified		Unclassified	Unclassified	UL

Final Report**Title: Creep Behavior of Solid Solution Strengthened $Y_3Al_5O_12$** **Award Number: F49620-97-1-0097****For the period of: 2/14/97-8/31/97**

Submitted to:

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In recent years there has been a recognition of the potential of structural ceramics for use in advanced turbine engines. Compared to metals, ceramics have higher operating temperatures, lower density, superior wear resistance and chemical stability. Unfortunately, monolithic structural ceramics also have a low fracture toughness resulting in brittle failure. One method of providing increased fracture toughness is to incorporate the ceramics in composite structures. It is anticipated that the new advanced turbine engines will be required to operate at temperatures above 1400°C in oxidizing environments. As a consequence of the oxidative environment it is most favorable to employ composites consisting of an oxide reinforcement and oxide matrix. In addition, use at these elevated temperatures makes creep resistance an important consideration. It is anticipated that ceramic composites will require creep rates on the order of 10^{-7} s^{-1} or lower for extended service applications in the advanced heat engines. To achieve these creep rates and to maximize fracture toughness it is desirable to use a fiber reinforcement. The creep resistance of a fiber composite at elevated temperature is primarily due to the creep resistance of the reinforcing fibers. In order to maximize the creep resistance of the oxide fiber it is desirable and possible in many cases to use single crystal fibers such as sapphire. The use of a single crystal eliminates the intergranular deformation mechanisms of diffusional creep and grain boundary sliding.

The creep rate of a single crystal oxide at high temperatures in general is a function of the dislocation density, dislocation glide mobility and dislocation climb mobility. Consequently, single crystal oxides with a low dislocation densities and low dislocation glide/climb mobilities are desired for high creep resistance. It has been suggested that criterion for selecting such a creep resistance oxide is to look for an single crystal with a large Burger's vector. As the magnitude of the Burger's vector increases the dislocation density will decrease and both dislocation glide and climb will become more difficult leading to high creep resistance material. Oxides with complex crystal structures (e.g., $\text{Y}_3\text{Al}_5\text{O}_{12}$ and BeAl_2O_4), having large unit cells and consequently, a large Burger's vector have recently received attention as possible reinforcements in the oxide/oxide composites as a result of their high creep resistance. To further increase the creep

resistance of such oxides it is necessary to continue to decrease the dislocation glide and/or climb mobility. One possible method that has not been investigated at present in these oxides is through the formation of solid solution alloys. For the case of oxides two different possible solid solution alloy types are possible: i) isovalent (e.g., Fe^{+2} in MgO) and ii) aliovalent (e.g., Cr^{+3} in MgO). For the case of both isovalent and aliovalent impurities it is possible to retard dislocation glide through an elastic and/or electrostatic interaction of the dislocation with the impurity (and/or the charge compensating defect for the case of an aliovalent impurity). For the case of the aliovalent impurities it is also possible to retard dislocation climb by reducing the point defect concentration of the rate-controlling ion and hence, lower the diffusivity of the species which is controlling the creep rate. The use of aliovalent transition metal impurities (e.g., Fe^{+2}) whose concentration is a function of the oxygen partial pressure is particular useful since not only can it reduce the creep rate but, can also provide insight into the rate-controlling point defects and hence, the species for the case of a diffusion controlled process. The effects of adding both isovalent and aliovalent impurities have extensively studied in the past for the case of binary oxides (e.g., MgO and Al_2O_3) with the observation that the addition of an aliovalent impurity had a much greater effect on increasing the creep resistance than an isovalent impurity. However, for the case of single crystal ternary oxides with complex crystal structures and large Burger's vector such as $Y_3Al_5O_{12}$ the effects of adding isovalent or aliovalent impurities on the creep behavior are not known. In addition, the creep mechanism of pure single crystal $Y_3Al_5O_{12}$ at present is still uncertain.

It is the intent of this study to: i) provide fundamental insight into the creep mechanism of single crystal $Y_3Al_5O_{12}$ and ii) provide a method of increasing the creep resistance of oxide single crystals which are under consideration as potential fiber reinforcements for use in elevated temperature oxide/oxide composites. At present, no information exists on the effects of isovalent or aliovalent impurities on the creep behavior of single crystal $Y_3Al_5O_{12}$.

Our work over the past five months since starting the project has focused on the creep behavior of pure single crystal $Y_3Al_5O_{12}$ to: i) confirm the limited existing data and ii) investigate the rate-controlling deformation mechanism. Figure 1 is the steady-state creep rate versus applied stress divided by the shear modulus for single crystal $Y_3Al_5O_{12}$ on a double logarithmic plot at a temperature of $1635^{\circ}C$. Data from Corman [1], Blumenthal et al. [2] and Karato et al. [3] (filled symbols) and the present investigation (open symbols) are shown. From Figure 1 three important points are noted. Firstly, the present data is in good agreement with the existing data. Secondly, the steady-state creep rate is nearly independent of orientation. All the data from the different orientations clusters about a common line. Thirdly, the stress exponent (slope of the line) for all the data is about 3.3.

The stress exponent (n) of 3.3 for pure single crystal $Y_3Al_5O_{12}$ suggests that the rate-controlling creep mechanism is a dislocation creep mechanism, most likely either a Nabarro [4] climb ($n=3$), where the creep rate is controlled by the climb of dislocations, or the Peierls [5] stress ($n=3$), where dislocation glide is rate-controlling. Blumenthal et al. [2] and Karato et al. [3] based on deformed microstructures and the observation that the activation energy for creep is a function of stress have suggested that the rate-controlling mechanism is the Peierls stress. However, none of the previous investigations compared the experimental creep data to the theoretical prediction for the Peierls stress mechanism. In Figure 1, the creep data from Corman [1], Blumenthal et al. [2] and Karato et al. [3] and the present study for pure single crystal $Y_3Al_5O_{12}$ at $T=1635^{\circ}C$ are compared with the theoretical predictions for the Nabarro and Peierls stress mechanisms. From Figure 1 it is observed that Nabarro's model predicts a creep rate that is about 10,000 times faster than the experimental rates whereas, the Peierls stress model predicts a creep rate about 200 times slower than experimentally observed. Thus, from Figure 2 it can be concluded that the creep behavior of pure single crystal $Y_3Al_5O_{12}$ is most likely controlled by the Peierls stress (dislocation glide).

A further proof of this is shown in Figure 2. Figure 2 is a creep curve for pure single crystal $Y_3Al_5O_{12}$ deformed at $T=1635^{\circ}C$ under an applied stress of 200 MPa. The data in Figure 2 are plotted as logarithm of instantaneous creep rate versus true strain. From

Figure 2 it is observed that the primary creep stage reveals an inverse transient, the creep rate increases with strain, before a steady-state creep rate is reached at about 3% true strain. The nature of the primary creep transient for pure single crystal $Y_3Al_5O_{12}$ suggests that it's creep behavior is indeed controlled by dislocation glide.

To further confirm that dislocation glide is the rate-controlling mechanism in pure single crystal $Y_3Al_5O_{12}$ two additional experiments were conducted: i) stress-change tests and ii) determination of the internal stress. The results of a typical stress reduction test are shown in Figure 3. The data in Figure 3 are plotted as logarithm of instantaneous creep rate versus true strain. The sample was deformed at a higher applied stress into steady-state, at which point the load was reduced (shown by the dashed line), and the creep rate at the reduced stress as a function of strain is recorded until steady-state creep behavior is achieved at the lower applied stress level. From Figure 3 it is observed that the initial creep rate after the stress reduction from 200 to 180 MPa is faster than the steady-state creep at 180 MPa. This type of creep transient is of the inverse type and is similar to that observed in metallic and ceramic alloys when dislocation glide is the rate-controlling process.

To determine the internal stress, σ_i , the stress, σ , was decreased in a series of equal steps (≈ 20 s between steps) and the creep rate was measured immediately following each stress reduction. The internal stress was determined from a plot of the reduced creep rate normalized by the original creep rate against $\Delta\sigma$, where $\Delta\sigma$ is the magnitude of the total stress change according to the relation:

$$\sigma_i = \sigma - \Delta\sigma_c \quad (1)$$

where $\Delta\sigma_c$ is the value of $\Delta\sigma$ when the reduced creep rate normalized by the original creep rate is equal to zero. The values of the internal stress at stress levels of 100, 200 and 300 MPa are shown in Figure 4. The data in Figure 4 are plotted as normalized internal stress versus applied stress. From Figure 4 it is observed the normalized internal stress is essentially independent of applied stress with values between 0.58 to 0.70.

Values of normalized internal stress approximately equal to unity correspond to creep controlled by dislocation climb in the steady-state creep region, whereas values of normalized internal stress controlled by dislocation glide in the steady-state regime are typically between 0.4 to 0.7. The internal stress values of the present study for pure single crystal $Y_3Al_5O_{12}$ are in closer agreement values for dislocation glide than dislocation climb, suggesting dislocation glide as the rate-controlling mechanism.

The nature of the creep transients upon initial loading and stress reduction tests (inverse), values of the normalized internal stress (≈ 0.6 to 0.7) and value of the stress exponent (≈ 3.3) suggest dislocation glide as the rate-controlling deformation mechanism. A comparison of the creep data for pure single crystal $Y_3Al_5O_{12}$ with the existing dislocation glide mechanisms suggest glide controlled by the Peierls stress mechanism. Transmission electron microscopy on deformed $Y_3Al_5O_{12}$ is currently underway to confirm the above suggestions. In addition, our next work will focus on creep testing of $Er_3Al_5O_{12}$ at $T=1635^{\circ}C$. $Er_3Al_5O_{12}$ forms a complete solid solution with $Y_3Al_5O_{12}$ with almost an identical melting point. This will be followed by testing of three solid solution alloys from the $Er_3Al_5O_{12}$ - $Y_3Al_5O_{12}$ system: i) 4 mol.% $Er_3Al_5O_{12}$ -96 mol.% $Y_3Al_5O_{12}$, ii) 30 mol.% $Er_3Al_5O_{12}$ -70 mol. % $Y_3Al_5O_{12}$ and iii) 50 mol.% $Er_3Al_5O_{12}$ -50 mol. % $Y_3Al_5O_{12}$ (These crystals have been already purchased). These results will allow us to determine if solid solution strengthening can be used as a method of increasing the creep resistance of $Y_3Al_5O_{12}$ and if so, what is the fundamental strengthening mechanism.

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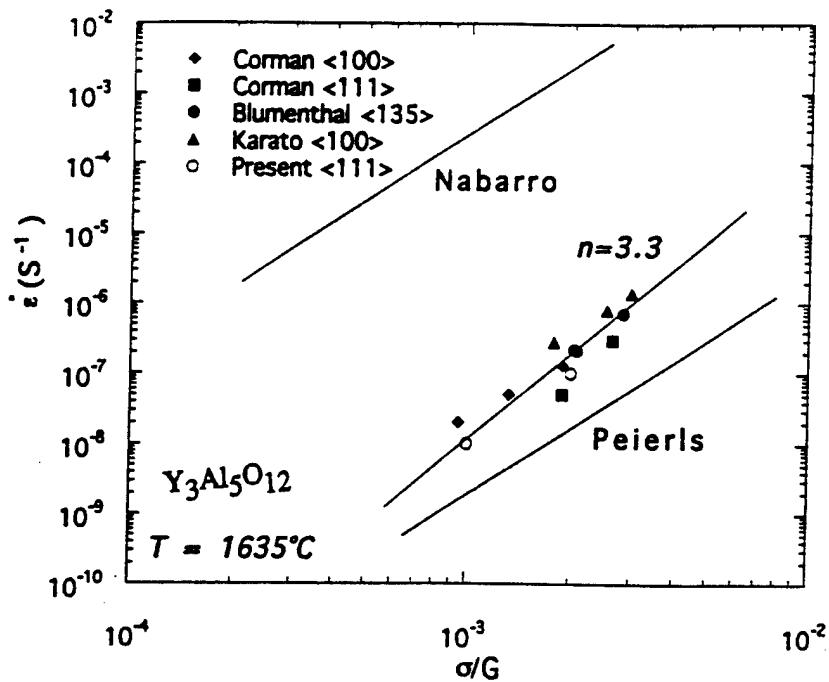


Figure 1. Steady-state creep rate versus modulus compensated stress for single crystal $\text{Y}_3\text{Al}_5\text{O}_{12}$ at a temperature of 1635°C . Data from Corman [1], Blumenthal et al. [2] and Karato et al. [3] (filled symbols) and the present investigation (open symbols) are shown. The Nabarro and Peierls predictions are shown for comparison.

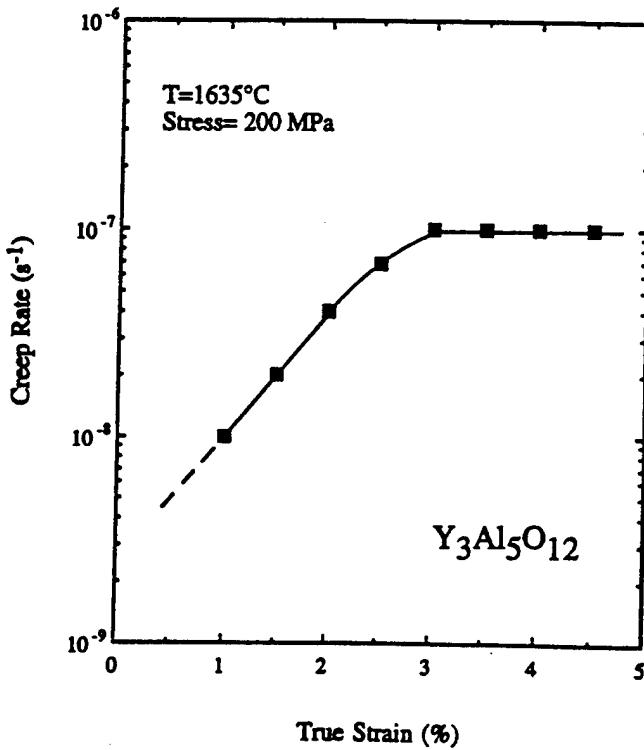


Figure 2. Inverse creep transient in $\text{Y}_3\text{Al}_5\text{O}_{12}$.

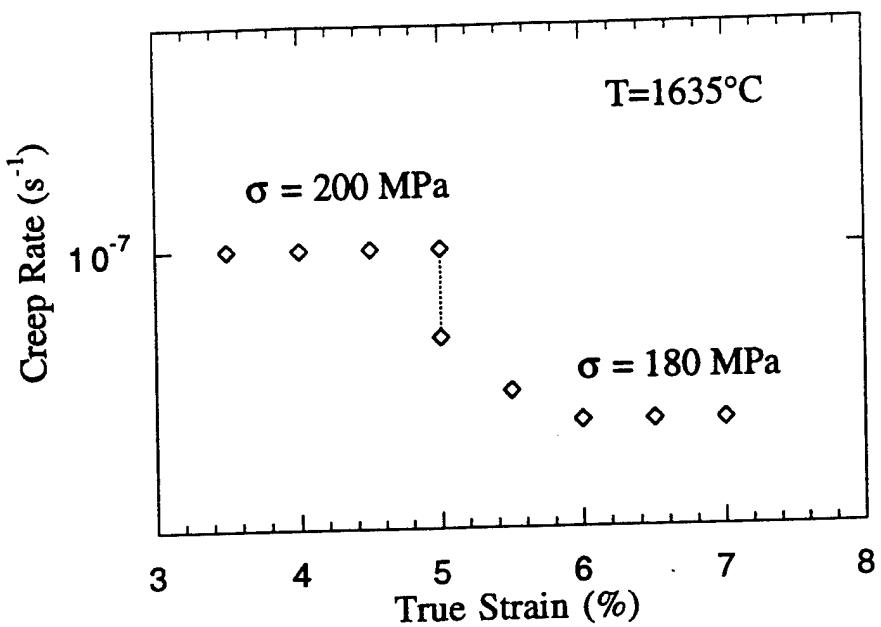


Figure 3. Typical results from a stress reduction test for pure single crystal $\text{Y}_3\text{Al}_5\text{O}_{12}$ deformed at $T=1635^\circ \text{C}$.

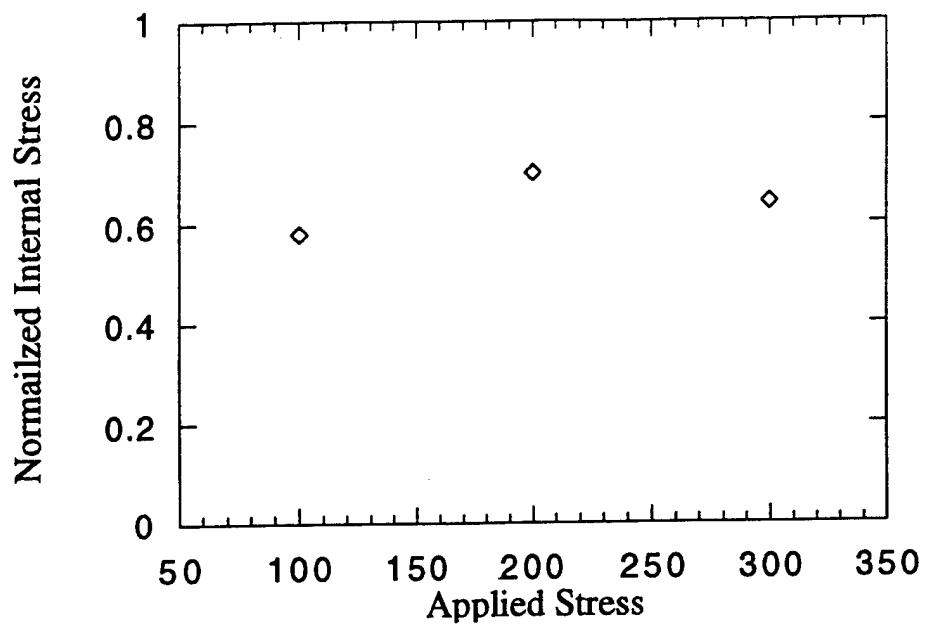


Figure 4. Normalized internal stress as a function of applied stress for pure single crystal $\text{Y}_3\text{Al}_5\text{O}_{12}$ deformed at $T=1635^\circ\text{C}$.